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PESTICIDE MONITORING SPECIAL STUDY NO. 44-0957-77
INVESTIGATION OF SUSPECTED NONOCCUPATIONAL HUMAN
INTOXICATION/CHLORDANE TERMITE TREATMENT
FORT MONMOUTH, NEW JERSEY
MAY - JUNE 1977

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Occupants of a quarters at Ft Monmouth, NJ became ill on the day the quarters were treated for subterranean termites, using a reported 1 percent chlordane water emulsion, by a contract exterminator. Chlordane intoxication was suspected as a cause of the illness.		
Blood serum samples from the occupants and a neighbor were analyzed for pesticide residues. No evidence of chlordane or its metabolites were found in the serum but normal levels of p,p'-DDE were found. (contd)		

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20.

Dust from the occupant's vacuum bag was analyzed for pesticide residues. Thlordane (approximately 30 ppm) was detected and trace amounts of diazinon and malathion were found.

A sample of the reported 1 percent chlordane water emulsion was analyzed for composition. The product was found to contain 0.544 percent chlordane, 0.76 ppm diazinon and 0.93 ppm malathion.

Although one of the quarters occupants manifested symptoms roughly compatible with chlordane overexposure, no evidence of increased body burden of chlordane or its metabolites was demonstrated in blood serum samples. No conclusions could be drawn from the presence of chlordane in the dust owing to a lack of available baseline data regarding probable or expected residual levels following routine treatments for subterranean termites.

Recommendations regarding termite inspection and treatment are made in the report.

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PESTICIDE MONITORING SPECIAL STUDY NO. 44-0957-77
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FORT MONMOUTH, NEW JERSEY
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ABSTRACT

Occupants of a quarters at Ft Monmouth, NJ became ill on the day the quarters were treated for subterranean termites, using a reported 1 percent chlordane water emulsion, by a contract exterminator. Chlordane intoxication was suspected as a cause of the illness.

Blood serum samples from the occupants and a neighbor were analyzed for pesticide residues. No evidence of chlordane or its metabolites were found in the serum but normal levels of p,p'-DDE were found.

Dust from the occupant's vacuum bag was analyzed for pesticide residues. Chlordane (approximately 30 ppm) was detected and trace amounts of diazinon and malathion were found.

A sample of the reported 1 percent chlordane water emulsion was analyzed for composition. The product was found to contain 0.544 percent chlordane, 0.76 ppm diazinon and 0.93 ppm malathion.

Although one of the quarters occupants manifested symptoms roughly compatible with chlordane overexposure, no evidence of increased body burden of chlordane or its metabolites was demonstrated in blood serum samples. No conclusions could be drawn from the presence of chlordane in the dust owing to a lack of available baseline data regarding probable or expected residual levels following routine treatments for subterranean termites.

Recommendations regarding termite inspection and treatment are made in the report.

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1. AUTHORITY.

- a. AR 200-1, Environmental Protection and Enhancement, 7 December 1973.
- b. AR 40-5, Health and Environment, 25 September 1974.

2. REFERENCE. Letter, AHDD-HE, US Army Medical Department Activities, Ft Monmouth, NJ, 11 May 1977, subject: Request for Technical Assistance.

3. PURPOSE. To determine if environmental exposure to a chlordane termite treatment at Ft Monmouth, NJ, on 2 May 1977 by a contract exterminator was related to the illness of the occupants of the treated quarters.

4. BACKGROUND.

a. On 2 May 1977, a quarters at Ft Monmouth, NJ, was treated for termites using a reported 1 percent chlordane water emulsion. This treatment was done by a contract operator who reportedly used normal treatment procedures including treatment of cinder block voids and injection.

b. On the same day, the occupants of the quarters became ill and chlordane intoxication was suspected. The occupants and their symptoms were:

(1) Three month old infant showing signs of pallor, irritability, labored breathing, convulsive jerking and stopped breathing. This reportedly was a doubtful seizure.

(2) Father, age 25, experienced vomiting.

(3) Mother, age 28, felt ill.

(4) Female dog, age 5, showed no signs of illness.

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(5) Female dog, age 4 months, was ill but had recently received rabies and distemper shots.

c. The following samples were requested and analyzed for pesticides:

(1) Serum samples from the infant, father, mother, both dogs and a neighbor. Serum samples were obtained 2 days after the occupants became ill. A control serum sample was taken from one of the Pesticide Monitoring Branch personnel.

(2) The contents of the household vacuum cleaner bag. A control vacuum cleaner bag was taken from one of the Pesticide Monitoring Branch personnel.

(3) A sample of the reported 1 percent chlordane water emulsion which was used for the treatment. This solution reportedly represented what was placed into the contract exterminator's spray tank; however, a sample of the actual spray tank contents was not received.

d. Replicate analyses were done on those samples for which there was sufficient sample available. Reagent blanks were run with each set of samples analyzed.

e. The methodology employed for the extraction and analysis of serum samples, vacuum cleaner dust samples and the chlordane water emulsion sample is described in Appendices A, B, and C, respectively.

5. FINDINGS.

a. Results of the serum analysis are given in Table 1 and the results of the vacuum dust analysis are given in Table 2. All samples were analyzed for the pesticides listed in Appendix D at the detection limits given.

b. The results of analysis of the reported 1 percent chlordane water emulsion sample were as follows:

chlordane - 5,438 ppm or 0.544 percent
diazinon - 0.76 ppm
malathion - 0.93 ppm

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TABLE 1. RESULTS OF ANALYSIS OF SERUM SAMPLES

Source of Sample	Pesticide Detected	Amount Detected (ppm)
Infant	None detected	
Mother Rep 1	p,p'-DDE	0.0064
Rep 2	p,p'-DDE	0.0068
Father	p,p'-DDE	0.0096
Neighbor	p,p'-DDE	0.0094
Young Dog	None detected	
Older Dog	None detected	
Control	p,p'-DDE	0.0419
	p,p'-DDT	0.0198

TABLE 2. RESULTS OF ANALYSIS OF VACUUM DUST SAMPLES
AMOUNTS DETECTED IN PPM

Pesticide Detected	Ft Mormouth Dust			Control
	Rep 1	Rep 2	Mean Rep 1/Rep 2	
chlordane	29.68	32.66	31.77	0
diazinon	1.55	1.77	1.66	0
malathion	0.19	0.18	0.19	1.05
p,p'-DDT	0	0	0	0.82
trans-chlordane	0	0	0	0.13
cis-chlordane	0	0	0	0.54

6. DISCUSSION.

a. Serum.

(1) None of the serum samples analyzed, including those from occupants of the treated quarters, contained any detectable levels of chlordane, or chlordane derived metabolites, such as heptachlor epoxide and oxychlordane.

(2) Several of the serum samples, including those from the mother, father, neighbor and control, did contain detectable levels of p,p'-DDE. The control serum sample also contained detectable amounts of p,p'-DDT. The finding of p,p'-DDE at the levels noted in the serum from the mother, father and neighbor was not unexpected or unusual. A number of studies^{1,2,3} have shown p,p'-DDE (and to a lesser extent, p,p'-DDT) to be a ubiquitous contaminant in human serum. The levels of p,p'-DDE and p,p'-DDT found in the control serum were considerably higher than the mean levels noted in the general population. However, the individual from whom the control serum was taken had a previous history of significant exposure to DDT in the course of his research work.

(3) In three of the serum samples, including the infant, young dog, and older dog, an unidentified, asymmetric peak was noted using electron-capture detection. This peak was in the retention time area of cis and trans-chlordane on the primary column used but not on the two alternate columns. The serum from the older dog contained the largest sized peak. Using flame photometric detection, a chromatographically similar unidentified, asymmetric peak was noted in the serum of the older dog. In an attempt to further characterize the unknown peak, the serum samples from the infant, young dog and older dog were subjected to a routinely used Florisil column cleanup and separation procedure. In all three samples, the unidentified, asymmetric peak was not recovered through the Florisil column procedure, probably indicating that the peak was considerably more polar than the pesticides routinely analyzed for by our laboratory (Appendix D).

¹ Dale, W. E., et al., "Chlorinated Insecticides in Human Serum," *Tox. and Appl. Pharmacol.*, 8 (2):337 (1966)

² Warnick, S. L., "Organochlorine Pesticide Levels in Human Serum and Adipose Tissue. Utah-Fiscal Years 1967-71," *Pestic. Monit. J.*, 6(1):9-13 (1972)

³ Wyllie, J., J. Gabica, and W. W. Benson, "Comparative Organochlorine Pesticide Residues in Serum and Biopsied Lipoid Tissue: A Survey of 200 Persons in Southern Idaho - 1970," *Pestic. Monit. J.*, 6(2):84-88 (1972)

(4) In all of the serum samples analyzed, including the control, several middle to late-eluting unidentified peaks were noted using flame photometric detection (peaks were absent using electron-capture detection). These peaks were not recovered through the Florisil column procedure described above in paragraph (3). The peaks may have represented phospholipids present in the serum⁴.

b. Vacuum Bag Dust.

(1) The chlordane residues in the dust from the quarters at Ft Mornmouth are significantly different from the control dust chlordane residues. The chlordane from the quarters showed characteristics of resulting from a recent application, i.e., the chlordane chromatographic elution pattern resembled the pattern for technical chlordane. However, the chlordane from the control dust showed signs of breakdown, i.e., only traces of the two major components of technical chlordane (cis and trans-chlordane) were present. The levels of chlordane found in the quarters appears to be significant. Assuming that the quarters were vacuumed the day of the treatment, or shortly thereafter, the levels found indicate that chlordane probably entered the quarters as a result of the treatment. Since the normal procedures for termite treatment does not include any application inside the quarters, the chlordane in the dust is remarkable.

(2) The presence of the organophosphorus pesticides, diazinon and malathion, in the vacuum bag dust was an unexpected finding since no recent applications of these pesticides in or near the quarters had been reported. Subsequent analysis of the chlordane water emulsion treatment solution (see discussion of this analysis in paragraph c below) indicated the presence of diazinon and malathion in the treatment solution; therefore, the finding of diazinon and malathion in the vacuum dust may be possibly due, at least in part, to the contaminated treatment solution. Another possible source of the diazinon and malathion in the vacuum dust may be the presence of a small amount of residual solution of the two pesticides in the spray tank which was mixed with and applied along with the chlordane treatment solution. The presence of diazinon and malathion in the vacuum dust, at the levels found, were probably not relatable to the illness of the occupants, especially since much higher levels (5X) of one of the organophosphorus pesticides, malathion, were found in the control vacuum bag dust [see discussion of control vacuum dust in paragraph (3) below].

⁴ Personal communication, Dr. C. C. Roan, US Army Environmental Hygiene Agency, Pest Management and Pesticide Monitoring Division (1977)

(3) Although the levels of malathion in the control are significantly higher than the quarters sample, this may be explained by the recent use of the control vacuum to clean a car which had been used to move plants and shrubs.

c. Chlordane Water Emulsion.

(1) Analysis of the chlordane water emulsion used in the treatment of the quarters indicated a concentration of 0.544 percent chlordane. The chlordane present in the water emulsion was qualitatively identical to an authentic technical chlordane standard; however, concentration of chlordane found was only about one-half of the reported 1 percent value. The reasons for this discrepancy between reported and experimentally determined chlordane concentration values were not investigated in this study. At least a part of the discrepancy was probably due to experimental error since the concentration of chlordane determined in the water emulsion was based only on a single weighing and analysis.

(2) The chlordane water emulsion sample also contained detectable quantities of two commonly used organophosphorous pesticides, diazinon and malathion. Although unexpected, the findings of low concentrations (i.e., less than 1.0 ppm) of diazinon and malathion in the treatment solution is most likely not relatable to the illness of the occupants. The chlordane treatment solution could have become contaminated with diazinon and malathion in a couple of ways: (a) use of rinse water from previous applications of diazinon and malathion as a diluent, and (b) use of equipment and/or utensils for dilution and mixing which had been previously used for diazinon and malathion. As mentioned previously in paragraph (2) of the discussion on vacuum bag dust results, the presence of malathion and diazinon in the dust may have possibly been due to the contaminated chlordane water emulsion treatment solution.

7. CONCLUSIONS.

a. Although the three month old infant demonstrated symptoms roughly consistent with chlordane overexposure, no evidence of increased body burden of chlordane or its metabolites could be elicited in blood serum samples analyzed for pesticide residues obtained from quarters occupants. The presence of p,p'-DDE at the levels found in serum of two of the occupants, the mother and father, was not unusual, and was reflective of the DDE serum levels present in the general population.

b. The presence of chlordane in the dust (qualitatively similar to technical chlordane) indicated a recent source of chlordane exposure. Although the levels of chlordane appear to be high, no conclusion as to the toxicological significance of these levels can be drawn owing to the absence of available baseline data regarding probable or expected residual levels following routine chlordane treatments for subterranean termites.

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c. The chlordane water emulsion treatment solution was qualitatively identical to authentic technical chlordane, although its concentration was only about one-half of that reported by the contract exterminator. The treatment solution also contained low levels of diazinon and malathion, a finding that may have been related to the presence of these pesticides in the vacuum dust from the treated quarters.

8. RECOMMENDATIONS.

a. All pesticide applications for subterranean termite control should be made in accordance with Sections 8.1.4 and 8.1.5 of TM 5-632, Military Entomology Operational Handbook, December 1971.

b. To minimize inadvertent or accidental acute exposure to chlordane, it is recommended that all occupants of quarters be removed during subterranean termite treatments and for a period of 24 hours following the treatments.

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APPENDIX A

PROCEDURES USED FOR ANALYSIS OF HUMAN SERUM

1. EXTRACTION OF SAMPLES.

a. The extraction of human serum samples was carried out using the procedures described in the US Environmental Protection Agency (USEPA) manual, Analysis of Pesticide Residues in Human and Environmental Samples.⁵

b. The only modification to the above referenced method used in this study involved rotation of serum samples at 35 rpm for 4 hours, instead of 50 rpm for 2 hours.

c. Definitive sample extract volumes for gas chromatographic analysis were 1.0 ml.

2. CLEANUP OF SAMPLES.

a. Several serum samples were subjected to a Florisil column cleanup and separation procedure. Details of the Florisil column procedure have been described in a previous special study.⁶

3. GAS CHROMATOGRAPHIC ANALYSIS OF SAMPLES.

a. Each human serum sample was analyzed using two gas chromatographic detector systems. The detector systems used were: (1) electron-capture, and (2) flame photometric (phosphorus mode).

⁵ Analysis of Pesticide Residues in Human and Environmental Samples, J. F. Thompson, ed., Pesticides and Toxic Substances Effects Laboratory, National Environmental Research Center, USEPA, Research Triangle Park, NC, Section 5A(3)a, (1974)

⁶ Pesticide Monitoring Special Study No. 44-0131-77, Pesticide Recovery Studies for Evaluation of Department of the Army Pesticide Monitoring Program Soil and Sediment Analysis Methodology. Part I. Determination of Pesticide and Polychlorinated Biphenyl Recoveries from Soil Extracted Immediately Following Fortification. October-December 1976. National Technical Information Service, AD-A035 782/2GA, 21 pp. (1976)

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b. Routine operating parameters used with each detector system are listed below:

(1) Electron-capture.

(a) Gas chromatograph. TRACOR® 560 digital.

(b) Detector. TRACOR high-temperature Ni⁶³ electron-capture with free-standing linearizer.

(c) Gas chromatographic columns used:

1.5% OV-17/1.95% QF-1 (Col 1)

5% OV-1 (Col 2)

5% OV-210 (Col 3)

(d) Recorder. Honeywell Electronik® potentiometric strip chart (1 mV) - Chart speed 0.5 in/min.

(e) Routine operating temperatures.

Oven: 196°C - 200°C (Col 1 and Col 2); 180°C (Col 3)

Detector: 300°C

(f) Carrier gas flow: all columns 50-60 ml/min (95% Argon - 5% Methane).

(2) Flame Photometric (Phosphorus Mode).

(a) Gas chromatograph. TRACOR MT-222, equipped with glass-lined injection ports.

(b) Detector. TRACOR flame photometric detector operating in phosphorous mode with 526 nm optical filter.

(c) Gas chromatographic columns used:

3% OV-1 (Col 1)

1.5% OV-17/1.95% QF-1 (Col 2)

(d) Recorder. Honeywell Electronik potentiometric strip chart (1 mV) - Chart speed 0.5 in/min.

* TRACOR is a registered trademark of Tracor Inc., Austin, TX.

® Electronik is a registered trademark of Honeywell Inc., Industrial Division, Fort Washington, PA 19034.

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(e) Routine operating temperatures.

Oven: 200°C (Col 1 and Col 2)
Injection port: 245°C
Detector: 320°C

(f) Carrier gas flow both columns, 50-60 ml/min nitrogen.

(g) Detector gas flow: hydrogen - 50 ml/min
air - 90 ml/min

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APPENDIX B

ANALYSIS OF DUST PROCEDURES

1. The analysis of the vacuum dust sample was carried out following Analysis of Pesticide Residues in Human and Environmental Samples⁵ with the following modifications:

- a. Two grams of dust were used to lower the limits of detectability.
- b. Alumina cleanup was not used.
- c. Extract definitive volume = 10 ml.

2. One sample and one replicate of the sample were analyzed along with a control sample from the authors vacuum bag.

3. Gas-Liquid Chromatography (GLC) was used to analyze the pesticide residues utilizing Electron Capture, Electrolytic Conductivity and Flame Photometric Detectors. The following GLC operating parameters were used:

- a. TRACOR MT-222 Gas Chromatograph.
 - (1) Electron Capture Detector.
 - (a) Ni⁶³ High temperature
 - (b) Operating temperature 275°C
 - (2) Carrier gas = 95% Argon/5% Methane.
 - (3) Injector.
 - (a) Glass lined.
 - (b) Operating temperature 250°C

⁵ Analysis of Pesticide Residues in Human and Environmental Samples, J. F. Thompson, ed., Pesticides & Toxic Substances Effects Laboratory, National Environmental Research Center, USEPA, Research Triangle Park, NC, (Revised December 1974)

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(4) Columns.

(a) 1.5% OV-17/1.95% QF-1 on 80/100 mesh Gas-Chrom Q. 6 ft x 1/4 in o.d.
"U" shaped. Oven temperature = 200°C. Gas flow = 65 ml/min.

(b) Three percent OV-1 on 100/120 mesh Gas-Chrom Q. 6 ft x 1/4 in o.d.
"U" shaped. Oven temperature = 185°C. Gas flow = 50 ml/min.

(5) Electrolytic Conductivity Detector.

(a) Halogen mode.

(b) Pyrolysis oven temperature = 880°C

b. TRACOR MT-220 Gas Chromatograph.

(1) Flame Photometric Detector.

(a) Phosphorous mode.

(b) Detector temperature - 200°C

(2) Carrier gas = N₂

(3) Injector.

(a) Glass lined.

(b) Operating temperature = 250°C.

(4) Columns.

(a) Three percent OV-1 on 100/120 mesh Gas-Chrom Q. 6 ft x 1/4 in o.d.
"U" shaped. Oven temperature = 200°C. Gas flow = 60 ml/min.

(b) Four percent SE-30/6 percent QF-1 on 80-100 mesh Gas-Chrom Q. Oven
temperature = 200°C. Gas flow = 60 ml/min.

APPENDIX C

PROCEDURES USED FOR ANALYSIS OF CHLORDANE WATER EMULSION

1. APPARATUS AND MATERIALS.

a. Glassware and Balances.

- (1) Volumetric flasks - 100 ml
- (2) Volumetric pipets - 1 ml, 9 ml
- (3) Disposable Pasteur pipets - 9 in
- (4) Disposable micropipets - 25 μ l
- (5) Glass weighing boats
- (6) Tared beakers - 250 ml
- (7) Mettler® M5 analytical balance

b. Solvents and Standards.

- (1) Acetone - nancgrade
- (2) Benzene - nancgrade
- (3) Technical chlordane standard - analytical reference grade

2. PREPARATION OF SAMPLES FOR GAS CHROMATOGRAPHIC ANALYSIS.

a. One gram of chlordane water emulsion (stated to be 1 percent concentration) was weighed into a 100 ml volumetric flask and acetone added to volume. The mixture was shaken vigorously for several minutes and allowed to set at room temperature for 2 hours.

b. The mixture was then serially diluted 1:100 and 1:10 (1:1,000 total dilutions) with benzene to achieve an appropriate concentration for electron-capture gas chromatographic analysis, i.e., 10 ng chlordane water emulsion/ μ l or 100 pg actual chlordane/ μ l (based on a 1 percent solution).

c. The stock mixture prepared in paragraph a above (e.g., 1 g chlordane water emulsion/100 ml acetone) was also screened using flame photometric detection.

® Mettler is a registered trademark of Mettler Instrument Corporation, PO Box 100, Princeton, NJ 08540.

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3. GAS CHROMATOGRAPHIC ANALYSIS PARAMETERS.

a. Electron-capture Detection.

- (1) Gas chromatograph. TRACOR 560 digital.
- (2) Detector. TRACOR high temperature Ni⁶³ electron capture with free standing linearizer.
- (3) Gas chromatographic column used:
1.5% OV-17/1.95% QF-1
- (4) Recorder. Honeywell Elektronik potentiometric strip chard (1 mV).
Chart speed 0.5 in/min.
- (5) Routine operating temperatures.
 - (a) Oven: 196°C
 - (b) Detector: 300°C
- (6) Carrier gas flow: 60 ml/min (95 percent Argon - 5 percent Methane).

b. Flame Photometric (Phosphorus Mode) Detection.

- (1) Gas chromatograph. TRACOR MT-220, equipped with glass lined injection ports.
- (2) Detector. TRACOR flame photometric detector operating in phosphorous mode with 526 nm optical filter.
- (3) Gas chromatographic columns used:
 - (a) Three percent OV-1
 - (b) Four percent SE-30/6 percent QF-1
- (4) Recorder. Honeywell Elektronik potentiometric strip chart (/mV).
Chart speed 0.5 in/min.
- (5) Routine operating temperatures.
 - (a) Oven: 198°C
 - (b) Detector: 225°C

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(c) Injection Port: 225°C

(6) Carrier gas flow - both columns, 60 ml/min nitrogen.

(7) Detector gas flow: hydrogen - 50 ml/min
air - 95 ml/min

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APPENDIX D

LISTING OF PESTICIDES ANALYZED FOR AND LOWER LIMITS OF DETECTABILITY
OF THESE PESTICIDES IN HUMAN SERUM AND VACUUM DUST

Pesticide	Lower Limits of Detectability (ppm) Electron-Capture Detection*	
	Human Serum	Vacuum Dust
α -BHC	0.0004	0.003
β -BHC	0.0015	0.013
aldrin	0.0012	0.010
chlordane	0.0089	0.075
<u>cis</u> -chlordane	0.0012	0.010
<u>trans</u> -chlordane	0.0012	0.010
oxychlordane	0.0012	0.010
o,p'-DDD	0.0030	0.025
p,p'-DDD	0.0024	0.020
o,p'-DDE	0.0030	0.025
p,p'-DDE	0.0024	0.020
o,p'-DDT	0.0030	0.025
p,p'-DDT	0.0045	0.038
dieldrin	0.0018	0.015
endrin	0.0032	0.027
heptachlor	0.0005	0.004
heptachlor epoxide	0.0012	0.010

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Pesticide	Lower Limits of Detectability (ppm) Electron-Capture Detection*	
	Human Serum	Vacuum Dust
lindane	0.0006	0.005
methoxychlor	0.0012	0.100
mirex	0.0030	0.025
toxaphene	0.120	1.00
chlorpyrifos	0.0018 0.0060 (FPD)	0.015 0.100 (FPD)
ronnel	0.0015 0.0060 (FPD)	0.013 0.100 (FPD)
diazinon	0.0079 0.0048 (FPD)	0.065 0.080 (FPD)
malathion	0.0012 0.0075 (FPD)	- 0.125 (FPD)
methylparathion	0.0045 0.0045 (FPD)	0.038 0.075 (FPD)
parathion	0.0030 0.0052 (FPD)	0.025 0.088 (FPD)

* Lower Limits of detectability for organophosphorous pesticides using flame photometric detection (FPD) are also given as indicated.